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PATENT
Case 4233C3

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants:	Donald B. Appleby et al	:	Group Art Unit: 1211
Serial No.:	08/360,184	:	Examiner: E. White
Filed:	December 20, 1994	:	
For:	**Polyol Polyester Synthesis**		

SECOND DECLARATION UNDER 37 C.F.R. §1.608 OF SCOTT D. PEARSON

Assistant Commissioner for Patents
Washington, DC 20231

Dear Sir:

I, SCOTT D. PEARSON, declare that:

1. I am a co-inventor of and am familiar with the present Appleby et al U.S. patent application, and I am the Declarant of the First Declaration Under 37 C.F.R. §1.608 of Scott D. Pearson presented in this application.

2. From January 1989 through March 1990, a series of pilot plant runs of the continuous sucrose polyester production process, including those pilot plant runs discussed herein, were performed under my direction and control using the pilot plant located in Building 96 of The Procter & Gamble Company's Ivorydale plant. The pilot plant runs investigated

various parameters of the synthesis of sucrose fatty acid polyesters (commonly referred to as sucrose polyesters or FG) by reaction of sucrose and fatty acid methyl esters. During the pilot plant continuous process runs, the operation of the continuous sucrose polyester production equipment, sample collection, analysis of collected samples, and data preparation were performed under my direction and control in accordance with established procedures.

3. Subsequent to each pilot plant run, the data collected during the pilot plant run was analyzed by myself and others over a period of time, typically ranging from one to several weeks, in order to determine if any hypothesis and/or conclusions concerning refinement and/or improvement of the sucrose polyester reaction and/or the sucrose polyester continuous process could be made. Hypotheses and/or conclusions resulting from a pilot plant run were typically investigated further in additional laboratory and/or pilot plant runs.

4. During my employment with The Procter & Gamble Company, I have, from time-to-time, prepared and distributed bi-weekly and monthly reports accurately describing my activities, and/or activities of those under my direction and control, during the prior two week or monthly period which was the subject of the report. For each report, I accurately set forth therein the ending date for the prior two week or monthly period which was the subject of the report.

5. From March 27, 1989 to March 31, 1989, a pilot plant run of the continuous sucrose polyester production process was performed under my direction and control using the

Building 96 pilot plant. The March 27, 1989 to March 31, 1989 pilot plant run was designated as the P90327 pilot plant continuous process.

6. I have reviewed Exhibit 13 and confirm that it is an accurate copy of the P90327 Experimental Test Plan dated March 28, 1989, which Glen R. Wyness and I prepared on or about March 28, 1989. The P90327 Experimental Test Plan sets forth the conditions under which the P90327 pilot plant continuous process was to be run and the conditions under which the P90327 pilot plant continuous process was run from March 27, 1989 to March 31, 1989. As set forth in Exhibit 13, an objective of the P90327 continuous process was to examine the effects of milled sucrose on the continuous reaction of sucrose and fatty acid methyl esters. Specifically, in the second run of the P90327 continuous process, sucrose which had been milled in the presence of soap in a single pass through a mill was employed in the sucrose polyester reaction, and in the third run of the P90327 continuous process, sucrose which had been milled to a greater than normal degree by recycling through the mill for 2.5 hours was employed in the sucrose polyester reaction.

7. I have reviewed Exhibit 14 and confirm that it is an accurate copy of the P90327 Run Summary dated July 14, 1989, which I prepared on or about July 14, 1989. As set forth in Exhibit 14, page 1, the sucrose which had been milled in the presence of soap in a single pass through a mill had a particle size of approximately 21 microns, while the sucrose which was recycled through the mill for 2.5 hours had a particle size of approximately 16 microns. These

sucrose particle sizes are within the preferred polyol particle size ranges set forth at page 13, line 38 - page 14, line 2 of the Appleby et al application.

8. As further set forth in Exhibit 14, the P90327 pilot plant continuous process demonstrated that smaller sucrose particles were utilized more effectively in the first stage of the reaction than the larger sucrose particles. Particularly, the unreacted sucrose concentration, which is a measure of the extent of a reaction, was about 0.37% when 16 micron sucrose particles were used, while the unreacted sucrose concentration was about 0.93% when 21 micron sucrose particles were used (Exhibit 14, page 1), thereby demonstrating a faster reaction speed was obtained using the smaller sized sucrose particles, as described at page 13, lines 8-10 of the Appleby et al application. The conversion to octaester was also greater in the reaction employing the smaller sucrose particle size (Exhibit 14, pg. 1).

9. From April 24, 1989 to May 5, 1989, a pilot plant run of the continuous sucrose polyester production process was performed under my direction and control using the Building 96 pilot plant. The April 24, 1989 to May 5, 1989 pilot plant run was designated as the P90424 pilot plant continuous process.

10. I have reviewed Exhibit 15 and confirm that it is an accurate copy of the P90424 Experimental Test Plan dated April 20, 1989, which I prepared on or about April 20, 1989. The P90424 Experimental Test Plan sets forth the conditions under which the P90424 pilot plant continuous process was to be run and the conditions under which the P90424 pilot plant

continuous process was run from April 24, 1989 to May 5, 1989. As set forth in Exhibit 15, page 1, objectives of the P90424 continuous process included examining the effects of milled sucrose particle size on the continuous process and the effects of using different temperatures, namely 265°F and 285°F, in later stages of the reaction.

11. I have reviewed Exhibit 16 and confirm that it is an accurate copy of the Industrial Chemicals Product Development Biweekly Report which I prepared on or about May 31, 1989. As set forth in Exhibit 16, when the later stages of the P90424 pilot plant continuous process were run at a temperature of 265°F, which is in the range disclosed at page 18, lines 6-10 of the Appleby et al application, sucrose conversion to sucrose octaesters increased and the amount of burnt sucrose decreased as compared with reactions wherein the later stages of the process were run at a temperature of 285°F. Additionally, the P90424 continuous process further demonstrated that high octaester product was obtained when the sucrose was wet milled.

12. From June 5, 1989 to June 15, 1989, a pilot plant run of the continuous sucrose polyester production process was performed under my direction and control using the Building 96 pilot plant. The June 5, 1989 to June 15, 1989 pilot plant run was designated as the P90605 pilot plant continuous process.

13. I have reviewed Exhibit 17 and confirm that it is an accurate copy of the P90605 Experimental Test Plan dated June 2, 1989, which I prepared on or about June 2, 1989. The P90605 Experimental Test Plan sets forth the conditions under which the P90605 pilot plant

continuous process was to be run and the conditions under which the P90605 pilot plant continuous process was run from June 5, 1989 to June 15, 1989. As set forth in Exhibit 17, objectives of the P90605 continuous process included examining the effects of reduced soap levels and lower later stage temperatures in the continuous sucrose polyester reaction. The effect of various soap levels in the continuous reaction in combination with finely milled sucrose and lower temperatures in later stages of the reaction were also studied.

14. As set forth on page 3, lines 12-13 of Exhibit 17, under my direction and control a feed slurry comprising 2450 pounds of fatty acid methyl esters and 800 pounds of sucrose was prepared for use in the P90605 pilot plant continuous process. As set forth on page 3, lines 9 and 15 of Exhibit 17, for run 1A, 1100 pounds of the feed slurry (about 270 pounds sucrose and 530 pounds fatty acid methyl esters) and 300 pounds of soap were mixed. Based upon a molecular weight of 342 for sucrose and a molecular weight of 322 for potassium stearate soap, run 1A initially comprised about 423 moles of soap and about 360 moles of sucrose, or about 1.18 moles of soap per mole of sucrose, which was about 50% of our then normal soap level. As set forth on page 3, lines 20-21 of Exhibit 17, for run 1C, 1100 pounds of the feed slurry and 150 pounds of soap were mixed. Based upon the above-noted molecular weights, run 1C initially comprised about 211 moles of soap and about 360 moles of sucrose, or about 0.59 mole of soap per mole of sucrose, which was about 25% of our then normal soap level and within the range disclosed at page 15, lines 15-16 of the Appleby et al application. For run 1B, the feed slurry was used without addition of soap as set forth at page 3, lines 12-13, 22 and 25 of Exhibit 17.

15. As also set forth in Exhibit 17, page 1, the P90605 pilot plant continuous process also investigated the effect of using lower temperatures of about 260°F and 245°F during later reaction stages. These temperatures are within the ranges disclosed at page 18, lines 6-10 of the Appleby et al application.

16. I have reviewed Exhibit 18 and confirm that it is an accurate copy of the Industrial Chemicals Product Development Biweekly Report which I prepared on or about June 14, 1989. As set forth in Exhibit 18, the P90605 continuous process demonstrated that use of the 50%, 25% and 0% of the then normal soap levels, providing molar ratios of soap to polyol of 1.18:1, 0.59:1 and 0:1, resulted in high conversions in the sucrose polyester continuous reaction and that the amount of soap present in the reaction had a strong effect on the esterification reaction rate. The use of low levels of soap in the sucrose polyester reaction is disclosed at page 15, lines 4-24 of the Appleby et al application.

17. I have reviewed Exhibit 19 and confirm that it is an accurate copy of the Chemicals Product Development Biweekly Report which I prepared on or about June 28, 1989. As set forth in Exhibit 19, a run during the P90605 continuous process which employed a combination of a reduced soap level of 50% of the then normal soap level and a lower temperature of about 250°F, which is within the range disclosed at page 18, lines 6-10 of the Appleby et al application, in later reaction stages resulted in good octaester conversion of about 95%.

18. From September 25, 1989 to October 6, 1989, a pilot plant run of the continuous sucrose polyester production process was performed under my direction and control using the Building 96 pilot plant. The September 25, 1989 to October 6, 1989 pilot plant run was designated as the P90925 pilot plant continuous process.

19. I have reviewed Exhibit 20 and confirm that it is an accurate copy of the P90925 Experimental Test Plan dated September 25, 1989, which Gene P. Hawkins and I prepared on or about September 25, 1989. As set forth in Exhibit 20, one of the objectives of the P90925 pilot plant continuous process was to examine the effects of later reaction stage reduced temperatures on difatty ketone ("DFK") levels in the sucrose polyester continuous process by using temperatures of about 230°F in a later stage of the reaction. This temperature is within the preferred ranges disclosed at page 18, lines 6-10 of the Appleby et al application. Additionally, combinations of reduced soap levels and lower temperatures in the subsequent reaction stages were studied.

20. I have reviewed Exhibit 21 and confirm that it is an accurate copy of the Chemicals Product Development Division-Food Ingredients Monthly Report which I prepared on or about October 1, 1989. As set forth in Exhibit 21, by reducing soap levels in the P90925 continuous process, reaction rates were increased and high octaester conversions were maintained although lower temperatures of about 220°F were used in a later stage of the reaction, in accordance with the preferred later stage temperatures disclosed in the Appleby et al application at page 18, lines 6-10.

21. From February 5, 1990 to February 23, 1990, a pilot plant run of the continuous sucrose polyester production process was performed under my direction and control using the Building 96 pilot plant. The February 5, 1990 to February 23, 1990 pilot plant run was designated as the P00205 pilot plant continuous process.

22. I have reviewed Exhibit 22 and confirm that it is an accurate copy of the P00205 Experimental Test Plan dated January 22, 1990, which Gene P. Hawkins and I prepared on or about January 22, 1990. The P00205 Experimental Test Plan sets forth the conditions under which the P00205 pilot plant continuous process was to be run and the conditions under which the P00205 pilot plant continuous process was run from February 5, 1990 to February 23, 1990. As set forth in Exhibit 22, page 1, one objective of the P00205 pilot plant continuous process was to examine the effects of the fatty acid methyl ester to sucrose molar ratio on the sucrose polyester continuous reaction, wherein fatty acid methyl ester to sucrose molar ratios of about 14:1, about 12:1, about 10:1, and about 8.5:1, which correspond to molar ratios of ester to esterifiable sites on the polyol of about 1.75:1, about 1.5:1, about 1.25:1, and about 1.06:1, respectively, were employed. The latter two molar ratios are within the ranges disclosed at page 17, lines 8-10 of the Appleby et al application.

23. As further set forth in Exhibit 22, page 1, another objective of the P00205 pilot plant continuous process was to examine the effect of higher pressures on the sucrose polyester continuous reaction, wherein pressures of about 25 mm Hg and about 30 mm Hg in later stages of the reaction were studied. These pressures are within the ranges disclosed at page 18, lines

17-27 of the Appleby et al application. Temperatures of about 265°F and about 255°F in the first stage of the reaction were employed. Additionally, during the P00205 pilot plant continuous process, carbon dioxide was used as a sparging gas as is recommended at page 18, lines 22-25 of the Appleby et al application. As also set forth in Exhibit 22, the P00205 pilot plant continuous process also studied the effects of combinations of reduced soap to sucrose ratios, sucrose milling treatment and gas sparging, as well as the effects of combinations of reduced soap to sucrose ratios, selected ester:sucrose molar ratios and reduced temperatures.

24. From March 26, 1990 to April 6, 1990, a pilot plant run of the continuous sucrose polyester production process was performed under my direction and control using the Building 96 pilot plant. The March 26, 1990 to April 6, 1990 pilot plant run was designated as the P00326 pilot plant continuous process.

25. I have reviewed Exhibit 23 and confirm that it is an accurate copy of the P00326 Experimental Test Plan dated March 19, 1990, which I prepared on or about March 19, 1990. The P00326 Experimental Test Plan sets forth the conditions under which the P00326 pilot plant continuous process was to be run and the conditions under which the P00326 pilot plant continuous process was run from March 26, 1990 to April 6, 1990. As set forth in Exhibit 23, page 1, an objective of the P00326 pilot plant continuous process was to continue difatty ketone experimentation on the continuous pilot plant system, i.e., to investigate conditions for reducing DFK content in the sucrose polyester product.

26. As further set forth in Exhibit 23, page 1, during the P00326 pilot plant continuous process, fatty acid methyl ester to sucrose molar ratios of about 13.5:1 and about 9:1, which correspond to molar ratios of ester to esterifiable sites on the polyol of about 1.7:1 and 1.125:1, respectively, were studied. Additionally, during the P00326 pilot plant continuous process the use of carbon dioxide as a sparging gas was studied. The effects of combinations of gas sparging, selected ester:sucrose molar ratios and higher later reaction stage pressures of about 60 mm Hg were also studied in the P00326 pilot plant continuous process.

27. I have reviewed Exhibit 24 and confirm that it comprises accurate copies of pages 1 and 56-64 of a report entitled "DFK Experimental Summary" which I authored in or about June and July 1990. Exhibit 24 summarizes the investigations conducted during the P00205 and P00326 continuous processes and my conclusions therefrom. As set forth in the figure on page 56, sucrose ester product comprising greater than 70% octaester was obtained using ester to sucrose molar ratios of about 10:1, 9:1 and 8.5:1, which correspond with molar ratios of ester to esterifiable sites on the polyol of about 1.25:1, 1.125:1 and 1.06:1. These ratios are within the preferred ratios set forth at page 17, lines 8-10 of the Appleby et al application. As set forth in the top figure on page 58, the reaction rate for the reaction using the ester to polyol molar ratio of 9:1 (corresponding with a molar ratio of ester to esterifiable polyol sites of 1.125:1) significantly increased when combined with nitrogen sparging during the reaction, as set forth at page 17, lines 19-29 of the Appleby et al application.

28. As further set forth on page 57 of Exhibit 24, nitrogen sparging at about 0.8 lb/hr and 2.4 lb/hr was employed in combination with higher later stage reaction pressures of about 20 mm Hg and 60 mm Hg, respectively. These reactions resulted in sucrose ester product comprising about 85% octaester and about 80% octaester, respectively, after about 12 hours, as set forth in the top figure on page 58. These reactions therefore demonstrated that higher reaction pressures could be used in combination with increased sparging rates to provide acceptable reaction speeds as disclosed at page 18, lines 17-29 of the Appleby et al application.

29. I have reviewed Exhibit 25 and confirm that it is an accurate copy of the Industrial Chemicals Product Development Biweekly Report which I prepared on or about April 19, 1989. As set forth in Exhibit 25, in April 1989, experiments were performed in which the crude reaction mixture was filtered to remove unreacted sucrose when the unreacted sucrose level dropped to a concentration between about 1% and about 2%. The filtered reaction mixture contained sucrose ester product comprising about 95% octaester about 2 hours after filtration while an unfiltered control reaction mixture contained sucrose ester product comprising about 88% octaester about 4 hours after the time of filtration. Therefore, filtering resulted in faster reaction kinetics and highly esterified product as described in the Appleby et al application at page 16, line 10 - page 17, line 6.

30. In December 1989 and January 1990, a plug flow reactor comprising a packed column reactor was constructed for investigating the effect of higher pressures and methanol mass transfer in a later stage of the sucrose polyester reaction. I have reviewed Exhibit 26 and

confirm that it is an accurate copy of the Chemicals Product Development Division-Olestra Process Monthly Report which I prepared on or about January 1, 1990. As set forth in Exhibit 26, when the sucrose polyester synthesis reaction was conducted in the packed column using pressures of about 25 mm Hg at the top of the column and about 45 mm Hg at the bottom of the column, sucrose ester product comprising about 99% octaester was obtained in less than about 2 hours. By comparison, as set forth in Exhibit 26, continuous stirred tank reactors typically use pressures in the 1.0 to 3.0 mm Hg range to obtain similar conversions in comparable reaction times. Therefore, the packed column reactor results demonstrated it was possible to increase operating pressure through more efficient means of methanol removal. The use of a packed column reactor in a later stage of the sucrose polyester reaction is disclosed at page 21, lines 30-37 of the Appleby et al application. As further set forth in Exhibit 26, residual sucrose, catalyst and soap were filtered out of the crude reaction mixture which was fed to the packed column; this reduced the viscosity of the reaction mixture as disclosed at page 16, lines 20-24 of the Appleby et al application and resulted in improved methanol removal.

31. As set forth in my First Declaration, I have reviewed the Hawkins Declaration submitted herewith. As accurately described in the Hawkins Declaration, ¶¶ 10-12, and Exhibit 27, a series of experiments was performed on or about December 11, 1989 through January 8, 1990 by Mr. Hawkins under my direction and control to investigate the effect of using a packed column in a later stage of the sucrose polyester reaction. As indicated in the Hawkins Declaration, ¶12, and pages 8-9 of Exhibit 27, reaction pressures of from 15 mm Hg to 100 mm

Hg at the top of the packed column reactor were investigated. These pressures are within the ranges set forth at page 18, lines 17-19 of the Appleby et al application.

32. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Respectfully submitted,

By: Scott D Pearson
SCOTT D. PEARSON

Date: 4/5/99

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